Depth of Cure of New Flowable Composite Resins

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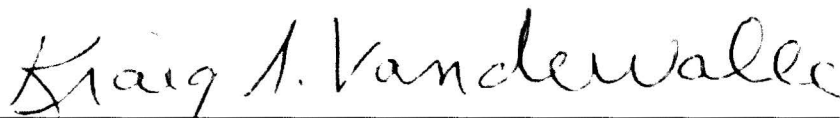Inaam A. Pedalino, BS, DDS

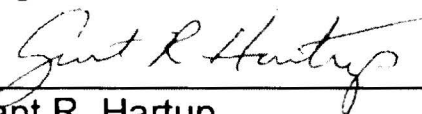Dunn Dental Clinic

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Depth of Cure of New Flowable Composite Resins

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DEDICATION

I would like to dedicate this work to my wonderful husband, Peter, who has been supporting me in all my endeavors since the day we met. His encouragement and undiminished love for the past 20 years has been my motivation. This thesis is also dedicated to my four children Anore, Afirah, Azra and Allya. Their patience, self-sacrifice and limitless support are beyond measure. Special thanks to Anore, in particular, for all she has done in stepping up to the plate, with both parents in residency programs, and helping to keep the family moving in the right direction. Thank you, my princesses, for allowing me to pursue my dreams, and now it is time to pursue yours.

ACKNOWLEDGEMENTS

ABSTRACT

Objectives: This study evaluated the depth of cure of Surefil SDR Flow (Dentsply), Grandio Flow (VOCO) and Venus Bulk Fill (Heraeus) and a conventional flowable composite, Revolution (Kerr) using bottom/maximum Knoop Hardness Number (KHN) ratios and the scrape technique (ISO 4049). **Methods:** Specimens were polymerized (Bluephase G2, Ivoclar) for 20 and 40 seconds at 0-mm distance. For KHN, five specimens were polymerized per flowable composite (shades A2, A3, and Universal for Venus) in 2-, 3-, 4-, 5-, and 6-mm-thick by 8-mm-diameter plastic molds. All specimens were stored for 24-hours at 37ºC in 95% humidity. KHN were determined from three measurements at each shade per thickness using a hardness tester (Leco). Maximum hardness was determined from the mean maximum KHN from the top surface of the 2-mm thick specimens polymerized for 40 seconds. The 4-mm thick mold was used first. If the bottom/maximum KHN ratio exceeded 80%, the next thicker mold was used, and if less than 80%, the next thinner mold was used. For the scraping technique, specimens were polymerized in a 14-mm by 4-mm diameter metal mold for 20 and 40 seconds at 0-mm distance. Uncured resin was scraped with a plastic instrument and the remaining thickness was measured with a micrometer and divided by two. Scrape-test data and the 4-mm specimens were analyzed with ANOVA/Tukey (alpha=0.05). **Results:** In general, the depth of

cure using either the bottom/maximum KHN or the scrape technique: Venus ≥ SDR ≥ Grandio ≥ Revolution.

Conclusions: Venus Bulk Fill predictably exceeded the manufacturer's claim of a 4-mm depth of cure using both KHN ratios and the ISO 4049 scrape test at both 20 and 40 seconds of curing time.

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For the past twenty years, the use of composite resin restorations has been on the rise and in many geographical markets they have replaced amalgam restorations altogether (Lindberg, 2004). Composite restorations are preferred by most patients due to their esthetic appeal (Korkmaz, 2007).   However, composite resin restorations are more technique sensitive and difficult to manipulate, requiring more time to place compared to amalgam restorations (Ozgünaltay, 2005). Therefore, dental product manufacturers have attempted to perfect the characteristics and qualities of composites to make them more ideal esthetic restorative materials possessing strong fundamental restorative characteristics.

Since the 1980's when light-cured direct composite restorative materials hit the dental marketplace, dentists have been in search of a tooth-colored amalgam replacement. Ideally, it would be a color-stable composite restoration that could be easily placed using a bulk-fill technique with a short curing time.   The restoration would have minimal polymerization shrinkage with no microleakage or fracture concerns (Burgess, 2010; Ikeda, 2009; Lee, 2005).

This has not yet occurred due to the properties that exist in today's composites leading to polymerization shrinkage stress during the curing process and a limited depth of cure for composite materials.   The effects of shrinkage is greater

on larger increments of composites, and if the curing light cannot adequately reach deeper surfaces of the restoration, the uncured portion of material will affect the bonding of the material to tooth structure and therefore affect the quality and longevity of the restoration. The recommended placement depth of composites is generally in 2mm increments to assure adequate polymerization and limited shrinkage stress (Burgess, 2010; Ikeda, 2009; Lee 2005).

Modern composite materials have less shrinkage and more wear resistance than earlier composites. Other properties, such as mechanical strength, polishability, color stability, and resistance to chemical and moisture breakdown, have also improved over the years. With fillers, such as strontium glass, barium glass, quartz, borosilicate glass, ceramic, and silica added to the composite matrix, the working properties and functional characteristics, such as reduced shrinkage, are greatly improved (Chalifoux, 2010).

Flowable composites were introduced to the dental community in the late 1990's (Ikeda, 2009; Bayne, 1998). The advantage of flowable composite-based resins is their ability to flow easily into small dental preparations with undercuts or in areas that were difficult to access (Ikeda, 2009). However, research has proven that flowable composites, in fact, shrink more than conventional composites because they have less filler content and/or more resin (Braga, 2003).

Flowable composites are fabricated with small particle sizes similar to hybrid composites but with less filler allowing the increased resin to reduce the viscous nature of the material (Ikeda, 2009; Bayne 1998). They also exhibit low wear resistance (Ikeda, 2009).

Therefore, flowable composites have not been used in bulk to fill large cavity preparations. Flowable composite resins have been used as a base or liner. The concept of placing a flowable composite underneath a posterior composite restoration was proposed to allow for better marginal adaptation and thereby reduce microleakage and to counter the polymerization shrinkage stress of the overlying composite resin because of its higher elastic properties (Braga, 2003; Awliya, 2008). However, laboratory studies evaluating the efficacy of a flowable composite as a liner have been equivocal (Gomeç, 2005).

The curing of composite materials occurs through the production of free radicals from either chemical, heat- or light-sensitive components. A mixture of catalyst and base, in early composites, created free radicals to cure composite. Heat activation produced free radicals, and hence, polymerized composite. The creation of light-cured composites provided control over the curing of the material. The composite material could now be placed, shaped and fine-tuned prior to curing without worry of pre-mature polymerization (Chalifoux, 2010).

The degree of cure of visible light-activated composite resins is vital to the success of these materials. Although the degree of cure of the external surfaces of a light-cured composite resin can be assessed quite easily, it is the degree of cure of the internal surfaces of the resin that cannot be assumed or easily evaluated (Moore, 2008).

Several factors can influence the depth of cure of a resin material. The light intensity and exposure time are some of those factors. The wavelength of light, the irradiance and the scatter of light within the restoration dictate the depth of light penetration through a composite restoration (Powers, 2006). A longer exposure time of the composite resin to the light source will increase the degree of polymerization. Therefore, it is recommended that exposure time be increased for darker composite resin shades or more opaque materials (Jain, 2003; Moore, 2008; Rueggeberg, 1993).

Hardness of the external surface of the composite is not an indicator of the extent of polymerization at the internal surface (O'Brien, 2002). Generally, the tip of the light source is held within 1-2mm of the surface of the composite with a standard exposure time of 20 seconds and a resin depth of approximately 2mm. For darker, more opaque shades, a curing time of 40 seconds is often recommended due to influence of the transmission coefficient.

The degree of polymerization of a light-cured composite resin cannot be accurately assessed by the degree of cure of the external surface. The physical properties of a composite can be hampered if the material is not polymerized through and through.

Although most manufacturers will recommend a specific curing time for 2-mm increments, Moore, et al. found that only the lightest of the shades of composite resin they tested met the minimum 2-mm standard for the depth of cure utilizing the ISO criteria for the evaluation of hardness, which tends to overestimate the degree of polymerization (Moore, 2008).

Depth of cure is often assessed indirectly by measuring the hardness of a composite resin material at specified depths. Higher hardness values correlate with a more extensive polymerization (DeWald, 1987). Depth of cure can also be defined as 50 percent of the remaining thickness of the composite resin after the uncured portion has been scraped off (Fan, 2002).

The scraping technique is an indirect method of assessing the depth of cure and is considered the standard for measurement of depth of cure as listed in the ADA specification (ISO Standard 4049, 2009). This method of measuring depth of cure is considered one of the simplest in both technique and cost. It consists of scraping away the underlying soft composite material. The maximum thickness of

the cured materials is then assessed utilizing a micrometer. The values are recorded as the depth of cure.

The Knoop Hardness or microhardness test is another indirect method in which the depth of cure is calculated. This test is extensively used due to its accuracy and simplicity. It consists of an indentation made by a Knoop elongated diamond pyramid with a load not to exceed 1 Kgf. This test measures the top or maximum and bottom surfaces for hardness, calculating a ratio which is compared against an arbitrary minimum value of adequate cure of the bottom surface. Typically, values of .80 and .85 have been used as this arbitrary minimum value. Therefore, a composite's bottom surface should be at least 80 percent as hard as the top or maximum hardness for that material (Moore, 2008).

Several studies have found that the scraping method can result in exaggerated depths of cure values compared to those values attained through the hardness test (DeWald, 1987). Ferracane, et al (1985) found that although the scraping technique is relatively easy to perform, there are limitations to the test that should be considered, often resulting in an overestimation of adequate depth of polymerizations. Nevertheless, the scraping technique allows a valid comparison of the depth of cure of materials.

Depth of cure of flowable composites as compared to traditional composites has been studied. Jain et al (2003) found when controlled for shade, cure time and thickness, microhybrid resin composites had the greatest depth of cure and flowable resin composites had the least depth of cure.

Shrinkage creates stresses between the composite and the tooth surfaces which can create interfacial stresses and small gaps or voids leading to microleakage. This gap may vary from 1.67% to 5.68% of the total volume of the restoration and may be filled with saliva, which can lead to postoperative sensitivity and recurrent caries (Deliperi, 2010). Polymerization shrinkage stress is influenced by the restorative technique, the modulus of resin elasticity, polymerization rate and the ratio of bonded to unbonded surfaces known as the "C-factor" or configuration factor (Deliperi, 2010). Placing composite resin in 2mm increments and curing each increment independently can reduce the net effect of polymerization shrinkage (O'Brien, 2002; Powers, 2006).

Several companies now claim to allow bulk fill of their flowable composite in increments over 4 mm. Three of these unique flowable composites are Surefil SDR Flow (Dentsply Caulk), Grandio Flow (VOCO) and Venus Bulk Fill (Heraeus Kulzer).

Surefil SDR (Stress Decreasing Resin) reportedly has a unique chemical that possesses a polymerization modulator that controls the matrix formation and allows for a more relaxed network to form than in conventional light-cured polymerization. It purportedly decreases stress by up to 60% through its curing process that creates minimal stress as the material is forming the bonds of polymerization (www.surefilsdrflow.com).

The manufacturer states that because of Surefil SDR Flow's unique polymerization initiating process and optical properties, light transmission is enhanced. The company claims a bulk fill of 4mm and a curing time of 20 seconds.

Grandio Flow (VOCO) is a nanohybrid flowable composite. It reportedly has increased stability, better material handling, low polymerization shrinkage, and a favorable thermal expansion coefficient. It is being marketed as the first flowable composite strong enough for Class I and Class II restorations.

Traditionally, flowable composites have shrinkage rates of approximately twice that of universal restorative composites. Grandio Flow, according to the manufacturer, shrinks only 2.99% - the percent shrinkage of most traditional composites. Also, the depth of cure is advertised as 4.3 mm bulk-fill based on A2 shade and a 40 second cure time. (www.vocoamerica.com)

Preliminary studies of these two products seem to indicate that Surefil SDR and Grandio Flow have mechanical properties comparable to conventional restorative composites. (Bracho-Troconis, 2010; Dai, 2010; Koltisko, 2010; Reis, 2010) In an unpublished study by Koltisko, B et.al, (2010), polymerization stress was lower for Surefil SDR Flow than other resin composites investigated.   Other studies indicate that Grandio Flow's shrinkage stress and marginal adaptation were similar to conventional restorative resins.   One study by Korkimaz et.al (2007) found that utilizing Grandio Flow as a liner beneath a composite resin reduced microleakage in the restoration.

Venus Bulk Fill is the newest posterior flowable nanohybrid composite on the market, as of the time of this study proposal. As per the manufacturer, Venus Bulk Fill can be utilized as a base in Class I and Class II restorations and polymerized up to 4 millimeters in thickness within a 20 second curing time at an irradiance of greater than 550 mW/cm² (www.heraeusdentalusa.com).

This study aimed to shed light on some of the claims made by these three product manufacturers and contribute to our knowledge base of flowable composite materials. Depth of cure and curing time was evaluated for Surefil SDR Flow (Dentspy Caulk), Grandio Flow (VOCO) and Venus Bulk Fill (Heraeus

Kulzer) compared to a traditional flowable, Revolution (Kerr) utilizing an LED light curing unit.

II.   OBJECTIVE

The purpose of this study was to evaluate the photocurability of new flowable composite materials which claim dramatically increased depth of cure through two different techniques for measuring curing depth, the scrap technique which is considered the standard for curing depth assessment (ISO 4049) and the Knoop Hardness Ratio which is a commonly used measuring tool for curing depth

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The Null hypothesis was that there is no significant difference in photocurability of the flowable composites.

III.   MATERIALS AND METHODS

Photocurability of Surefil SDR Flow, A2 and A3 shades, Grandio Flow, A2 and A3 shades, Venus Bulk Fill, universal shade was compared to a popular flowable composite material, Revolution, in A2 and A3 shade.   See Figure 1.

An irradiance level of 1200 mW/cm^2 was utilized in order to represent a typical irradiance level of new curing lights that are available and commonly purchased today. The Bluephase G2 LED light curing system (Ivoclar Vivadent Inc.) was used in this study.

The Bluephase G2 achieves a broad emission spectrum of 360 nm to 540 nm and includes a high intensity of 1200 mW/cm^2. According to the manufacturer, the Bluephase G2 light is suitable for all light initiators due to its proprietary Polywave LED (www.ivoclarvivadent.us).

The light emission from the Bluephase G2 was analyzed with a spectrophotometer (Blue Light analytics, Halifax, Canada). The curing light was connected to a power cord to provide continuous, consistent operation. The emitted light was analyzed during a 20-second curing cycle and the following data was collected:

Mean irradiance - 1132 mW/cm2

Total energy density - 22.8 J/cm2

Spectral – 360 - 420 nm – 4.2 J/cm2

　　　　　 420 - 540 nm – 18.6 J/cm2

Each specimen was polymerized at distances of 0 millimeters utilizing a clamp to hold the light source. The curing time was set at 20 seconds and 40 seconds for each of the four composites. The depth of cure properties was evaluated under

two different testing methods, surface hardness and the scraping technique (ISO 4049).

For the surface hardness test, the specimens were prepared in an 8 millimeter diameter split plastic ring mold. The plastic ring mold consisted of depths of 2, 3, 4, 5, and 6 millimeters for each sample group. Each sample group was made up of 5 specimens each (n=5).

An extracted third molar with its crown sectioned mid-coronally and dentin exposed was set in a rectangular base of epoxy resin. This created a similar background for the curing of the composite as seen in vivo. See Figure 2.

A plastic strip was placed over the exposed dentin and the mold was placed individually on top of the plastic strip. The composite was injected into the mold, a plastic strip placed, and condensed with a glass slide to displace the excess resin. The glass slide was then removed and the specimens were exposed to the external light source accordingly. See Figure 3.

For each depth, one group was polymerized at 1200 mW/cm^2 for 20 seconds and one group was polymerized at 1200 mW/cm^2 for 40 seconds. See Figure 4. The

specimens were removed from the plastic ring molds (see Figure 5) and stored in a light-proof container at 37°C for 24 hours. The surface hardness of the specimens was evaluated for hardness at the respective depth utilizing a Knoop Hardness tester (Leco, LM300AT, St Joseph, MI) under a load of 200 grams for 10 seconds. See Figure 6.

Three measurements were taken from the bottom of each sample. These measurements were used to calculate a mean bottom to maximum Knoop Hardness Number (KHN) ratio per composite per distance. The composite specimen was determined to be cured at that depth if the bottom surface had a KHN greater than 80% of the maximum hardness. Maximum hardness was determined by 3 measurements taken from the top surface of the 2-mm specimens cured for 40 seconds. The mean KHN ratio and standard deviation for each composite material was then calculated. The 4 mm deep mold was used first. If the mean KHN ratio was greater than 80%, the 5 mm mold was then utilized; and likewise, the 6 mm deep mold was used as necessary. If the 4 mm deep mold resulted in a mean KHN ratio less than 80%, the 3 mm mold was utilized; and likewise, the 2 mm deep mold was used as necessary.

Specimen samples were also tested using the scraping technique (ISO 4049). The scraping technique is the ISO standard for dental resins. Five specimens for each respective group were created by injecting composite resin into 4 mm

diameter x 14 mm long metal molds. One group was polymerized at 1200 mW/cm^2 for 20 seconds and another group was polymerized at the same irradiance level for 40 seconds, both at a distance of 0mm.

The uncured resin was then scraped with a plastic instrument starting from the deepest point on the underside of the mold until polymerized resin was reached. See Figure 7. According to the ISO standard, the length of the remaining polymerized material was measured with a digital micrometer and divided by two. The mean depth of cure and standard deviation for each composite material was calculated, accordingly.

The compiled data was analyzed against manufacturers' claims of depth of cure as per both measuring techniques for depth of cure used in this study and described above.

One-way ANOVA/Tukey (alpha=0.05) was used to assess the data compiled with the Scrape Test and the Knoop Hardness data of the 4mm thick specimens for A2 and A3 shades for Surefil SDR Flow, and Grandio Flow with Revolution as our control flowable composite material. Venus Bulk Fill in its available Universal shade was also assessed utilizing the same parameters.

Figure 1 - Composites Used Throughout Experiment
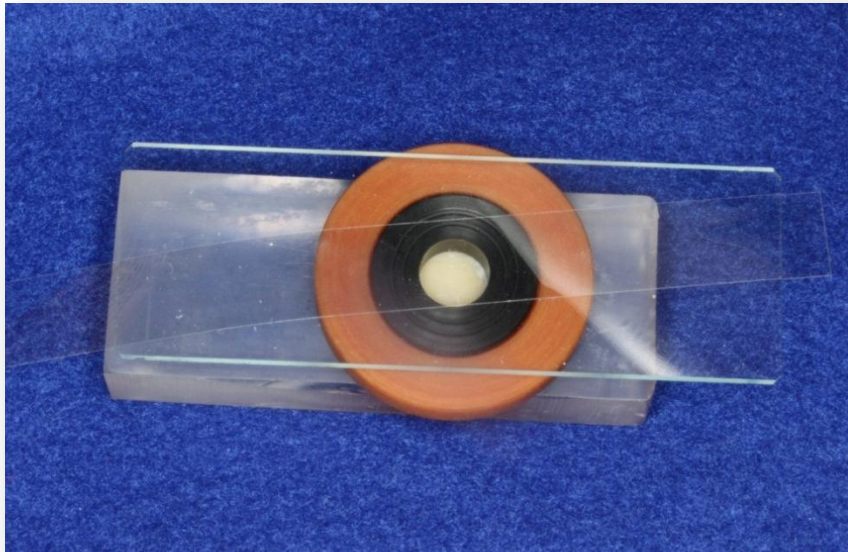

Figure 2 - Armamentarium

Figure 3 - Example of Experimental Set-up for Surface Hardness Test
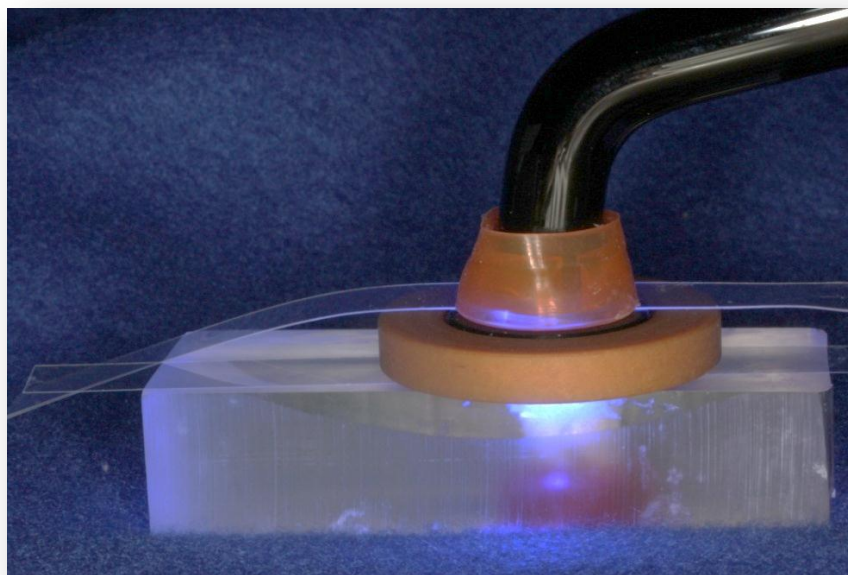

Figure 4 – Demonstration of Light Penetration Through Tooth Dentin

Figure 5 – Specimen of Composites Used for Surface Hardness Technique

Figure 6 – Knoop Hardness Tester

Figure 7 – Depth of Cure Assessment from Scrape Technique

IV. RESULTS

Depth of cure was evaluated using both the bottom/maximum Knoop Hardness Number and the scrape technique (ISO 4049). With either technique Venus Bulk fill showed a greater depth of cure than all the other flowable composites tested. See Table 1.

For shade A2 under the Knoop Hardness technique of depth of cure assessment, Surefil SDR Flow exceeded the 80% ratio of depth of cure at a thickness of 3mm for 20 seconds (89.6%) and 4mm at 40 seconds (91.3%) curing time. Grandio Flow was completely polymerized at depths of 2mm for 20 seconds (87.3%) and 3mm at 40 seconds (85.7%). Revolution in the shade of A2 exceeded the KHN ratio of 80% at thickness of 2mm for 20 seconds (89.3%) and 3mm at 40 seconds (81.3%) curing time.

For shade A3 under the Knoop Hardness technique of depth of cure assessment, Surefil SDR Flow exceeded the minimum 80% KHN ratio at 3mm thickness for both the 20 (81.4%) and 40 second (85.6%) cure time. Grandio Flow met this criterion at 3mm thickness for 40 seconds (81.4%) but did not meet this standard for 20 second curing time at a material thickness of 2mm or greater. Revolution exceeded the maximum hardness at 2mm for 20 seconds (86.6%) and 3mm for 40 seconds (84.3%).

Venus Bulk Fill, Universal shade, surpassed the maximum hardness, exceeding the 80% Knoop Hardness Ratio at 4mm of thickness cured for 20 seconds (89.3) and 5mm of thickness cured at 40 seconds (82.9).

The following results were obtained utilizing the scrape technique: For shade A2, Surefil SDR Flow completely polymerized 3.77 mm of material when cured for 20 seconds and 4.51 mm when cured for 40 seconds.   Grandio Flow was adequately cured at a depth of 3.01 mm when cured at 20 seconds and 3.56 at 40 seconds.   Revolution was the control parameter, polymerizing an average thickness of 2.88 mm when cured for 20 seconds and an average thickness of 3.24 mm when cured for 40 seconds.

Surefil SDR Flow, for shade A3, averaged a curing depth of 3.88 mm for 20 seconds of cure time and 4.52 mm for 40 seconds of cure time. Grandio Flow, at the same designated shade, cured 2.77 mm and 3.17 mm at curing times of 20 and 40 seconds, respectively. Revolution averaged a thickness of 2.42 mm for 20 seconds and 3.03 mm for 40 seconds when shade A3 was utilized.

Venus Bulk Fill, at its Universal shade, exceeded all other materials for depth of cure when assessed under the scrape technique.   Measurement averages of

4.88 mm and 5.07 mm thickness for 20 seconds and 40 seconds of curing time, respectively, were achieved with Venus Bulk Fill.

The percent Knoop Hardness ratios per composite, shade and curing time were regressed with a linear equation using data points on either side of 80% to determine the depth of cure in millimeters when the bottom surface of the composite had cured 80% of maximum. The depth of cure in millimeters based on percent Knoop Hardness ratios was compared to the scrape technique. The data is displayed in Table 2 and Table 3. The data was analyzed by a two-way ANOVA to examine the effects of composite or technique on depth of cure per shade, and curing time. Differences were found per composite and technique, however, there were significant interactions (p<0.05). The effect of technique on depth of cure was product and shade specific. The data was further analyzed with an unpaired t-test per shade, composite, and curing time. A Bonferroni correction was applied (alpha = 0.025) because multiple comparisons were made simultaneously. In the majority of groups there was no significant difference between the two techniques to determine depth of cure. The two techniques were found to be significantly correlated using a Pearson Correlation (p<0.001) with an R-squared of 0.78. See Figure 8.

Table 1: Bottom/Maximum Knoop Hardness Ratios

Flowable Composite	20-Second Curing Time Bottom/Maximum Knoop Hardness Ratios (st dev)										Scrape Test mm (st dev)	
	2 mm		3 mm		4 mm		5 mm		6 mm			
	A2	A3	A2	A3	A2	A3	A2	A3	A2	A3	A2	A3
Revolution	89.3* (2.9)	86.6* (5.8)	75.2 (2.2)	70.6 (1.9)	71.1 (1.8) a	51.9 (4.6) a					2.88 (0.03) a	2.42 (0.06) a
Grandio	87.3* (1.2)	75.5 (2.9)	79.8 (0.9)	69.1 (3.7)	67.1 (0.8) a	56.5 (2.5) ab					3.01 (0.04) a	2.77 (0.05) b
SDR			89.6* (3.0)	81.4* (1.9)	79.2 (1.2) b	59.6 (3.1) b					3.77 (0.19) b	3.88 (0.05) c
Venus					c 89.3* (3.9) c Universal		79.0 (2.4) Universal				c 4.88 (0.04) d Universal	
	KHN indicated with an asterisk (*) exceeded the minimum 80% bottom/max ratio. Groups with the same letter per column are not significantly different (p>0.05).											

| Flowable Composite	40-Second Curing Time Bottom/Maximum Knoop Hardness Ratio (st dev)										Scrape Test mm (st dev)	
	2 mm		3 mm		4 mm		5 mm		6 mm			
	A2	A3	A2	A3	A2	A3	A2	A3	A2	A3	A2	A3
Revolution			81.3* (5.9)	84.3* (3.1)	73.3 (2.0) a	60.1 (4.7) a					3.24 (0.11) a	3.03 (0.06) a
Grandio			85.7* (1.4)	81.4* (6.6)	76.6 (1.1) a	69.6 (2.0) b					3.56 (0.04) b	3.17 (0.04) a
SDR				85.6* (1.7)	91.3* (1.5) b	76.7 (2.4) c	77.3 (2.0)				4.51 (0.11) c	4.52 (0.25) b
Venus					b 90.9* (2.4) d Universal		82.9* (6.3) Universal		78.2 (2.7) Universal		d 5.07 (0.02) c Universal	
	KHN indicated with an asterisk (*) exceeded the minimum 80% bottom/max ratio. Groups with the same letter per column are not significantly different (p>0.05).											

Table 2. Depth of Cure in Millimeters

Curing Time	Technique	Depth of Cure Millimeters (st dev)						
		Revolution		Grandio		SDR		Venus
	Technique	A2	A3	A2	A3	A2	A3	Universal
20 Seconds	Scrape	2.88 (0.03) a	2.42 (0.06) a	3.01 (0.04) a	2.77 (0.05) a	3.77 (0.19) a	3.88 (0.05) a	4.88 (0.04) a
	% KH Ratios	2.66 (0.12) b	2.31 (0.30) a	2.98 (0.09) a	1.18 (0.63) b	3.94 (0.07) a	3.06 (0.09) b	4.81 (0.30) a
		Groups with the same letter per column are not significantly different (P>0.012)						

Table 3. Depth of Cure in Millimeters

Curing Time	Technique	Depth of Cure Millimeters (st dev)						
		Revolution		Grandio		SDR		Venus
	Technique	A2	A3	A2	A3	A2	A3	Universal
40 Seconds	Scrape	3.24 (0.11) a	3.03 (0.06) a	3.56 (0.04) a	3.17 (0.04) a	4.51 (0.11) a	4.52 (0.25) a	5.07 (0.02) a
	% KH Ratios	3.18 (0.37) a	3.17 (0.12) a	3.63 (0.07) a	3.14 (0.36) a	4.48 (0.5) a	3.66 (0.18) b	5.61 (0.25) b
		Groups with the same letter per column are not significantly different (P>0.012)						

Figure 8. Depth of Cure Correlation
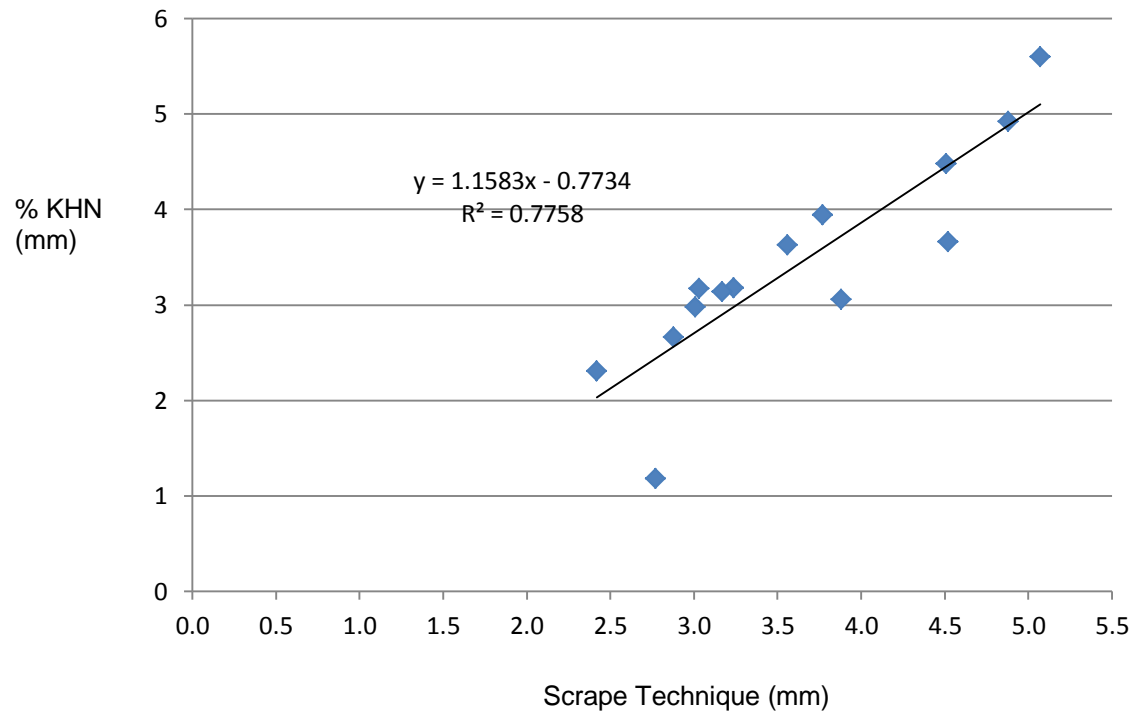
V. DISCUSSION

Curing time and depth of cure are important factors in light-cured composite resins. Placement and curing of resins in smaller increments, approximately 2mm or less, is often advised in order to assure complete polymerization of the composite (Ikeda 2009).

It has been proven that inadequate polymerization reduces the physical properties of the resin (Moore 2008). With polymerization comes polymerization shrinkage, ranging from 2.44 - 6.79% (Napoles, 2009, Lien, 2010) for methacrylate-based resins. Polymerization shrinkage can lead to stress at the interface between the composite and tooth structure and weaken that bond (Lee, 2005; Lindberg, 2004) and lead to adhesion failure or microleakage and increase the possibility of postoperative sensitivity, pulpitis and recurrent or secondary caries (Sadeghi, 2009).

A rapid polymerization and a higher degree of conversion increase the shrinkage stress of the composite restoration (Lindberg, 2004). Some have theorized ways to reduce this shrinkage stress such as using soft-start and pulse-curing methods as well as utilizing a flowable composite as a bottom layer to line a composite restoration.

On the other hand, a high degree of conversion is important in obtaining good mechanical properties and biocompatibility. A high degree of conversion is directly correlated to the total irradiance reaching the material, which is dependent on the curing unit and the distance between the curing tip and the composite resin (Lindberg, 2004).

Flowable composite resins are reported to have weaker mechanical properties, such as flexural strength and wear resistance, than conventional composite resin materials. Therefore, the use of flowable composites has been emphasized more in low-stress applications, such as sealants, preventive resin restorations and Class III and V restorations (Ikeda, 2009). Flowable composite resins have been suggested for use as liners due to their low viscosity, low elastic modulus and wettability (Korkmaz, 2007; Sadeghi, 2009).

Flowable composites can be easily injected into small cavities to improve adaptation to the cavity wall as opposed to conventional restorative composites which have a higher viscosity. There have been contradictory and inconclusive results as to whether this technique improves the marginal seal of the restoration resulting in decreased microleakage (Braga, 2003).

However, recently, manufacturers have introduced flowable composite resins of high filler content (Awliya, 2008; Ikeda, 2009). The claims are that the filler

content and polymerization shrinkage compare to conventional hybrid composites but with decreased viscosity and, hence increased "flow-ability", and increased depth of cure (www.vocoamerica.com). It is projected that their use in restorative treatments will include restoration of larger and deeper cavity preparations with increased thickness (Ikeda, 2009).

Three of these new flowable composite resins are Surefil SDR Flow by Dentsply, Grandio Flow by VOCO, and Venus Bulk Fill by Heraeus Kulzer. This study evaluated the claims made by these three manufacturers with respect to the depth of cure and the curing times for adequate polymerization of these unique materials.

Surefil SDR flow claims to be a low-stress flowable composite which can be bulk filled up to 4 mm increments. It contains a SDR patented urethane dimethacrylate resin which creates the reduction in polymerization shrinkage and stress. This SDR Technology, as it is referred to by the manufacturer, is a combination of a large molecular structured resin, SDR resin with a molecular weight of 849 g/mol, with a chemical moiety called a "polymerization modulator" chemically integrated into the center of the resin structure of the SDR resin monomer. With these features SureFil SDR flow claims to be the first posterior flowable base which can be bulk filled for use in Class I and II cavity preparations (www.surefilsdrflow.com).

The manufacturers of Surefil SDR flow site their own studies on depth of cure utilizing the ISO 4049 scrape technique. In their study, the depth of cure of SureFil SDR Flow, in its Universal shade, was measured against other flowable composites in shades ranging from Universal to A2, including Grandio Flow (A2), Revolution Formula 2 (A2), and Venus Bluk Fill (U). The restorative materials were light cured for 20 seconds in a stainless-steel mold utilizing a Spectrum 800 halogen light at light intensity of 500-550 mW/cm^2. The uncured underside was scraped away with a plastic spatula. The remaining thickness of material was then measured using a micrometer and divided by two to get the depth of cure measurement. According to the manufacture, SureFil SDR Flow was completely cured at 4 mm increment thickness (www.surefilsdrflow.com).

It is interesting to note their study indicates a curing depth of almost 5 mm incremental thickness with Venus Bulk Fill. Their data also indicates that Grandio Flow reached a depth of cure of 2.8 mm compared to Revolution curing at 2.5 mm thickness. These results were similar to the results achieved in this study.

Grandio Flow is a nano-hybid flowable composite with a high filler content of 80.2 w/w % and a resin portion that is up to 50% less than traditional flowable materials. The manufacturers of Grandio Flow claim a depth of cure of 3.5 mm

increments for 20 seconds for shade A2, and a depth of cure of 4.3mm at 40 seconds, verified by their research data utilizing the scrap technique (www.vocoamerica.com).

Venus Bulk Fill is marketed as a low stress flowable composite which enables 4 mm bulk fill with a 20 second curing time. It has self-adaptive handling properties for ease of placement in posterior cavity preparations. Two studies were sponsored by the manufacturer for depth of cure. One study utilized the ISO 4049 technique and evaluated SureFil SDR Flow and Venus Bulk Fill. The other study utilized the Knoop Hardness Ratio to compare the curing potential of 4 mm increments of Venus Bulk Fill to other composite materials, Filtek Supreme Ultra (3M ESPE), Filtek Supreme Plus (3M ESPE), and Venus Diamond (Heraeus). The first study, using the scrape test, showed no significant difference between SureFil SDR Flow and Venus Bulk Fill. The other study, using the Knoop Hardness test, showed that Venus Bulk Fill and Filtek Supreme Ultra were both capable of complete cure of 4 mm increments at 20 seconds (Heraeus 2011).

Generally speaking, the results compiled in this study are comparable to the overall results found in the above mentioned studies by the different manufacturers. In this study, there was a statistically significant difference between the materials studied. Therefore, this study's null hypothesis of no significant difference in photocurability of the flowable composites was rejected.

To review, depth of cure properties for four different flowable composites, SureFil SDR Flow, Grandio Flow, Venus Bulk Fill, and Revolution, which served as our control, were evaluated using two different testing methods, surface hardness (Knoop Hardness Number Ratio) and the scraping technique (ISO 4049). Data was compiled on curing times of 20 and 40 seconds as well as by shade. SureFil SDR Flow, Grandio Flow and Revolution were tested in their respective A2 and A3 shades. Venus Bulk Fill is currently available in Universal shade, and consequently evaluated in only that shade. For the surface hardness test, the composite materials were first assessed in molds 4 mm in thickness. If the bottom/maximum KHN ratio exceeded 80%, then a 5 mm mold was used. If not, a 3 mm mold was then used. This was continued for molds of 2 to 6 mm in thickness.

Data was compiled for each mold thickness in accordance with the above method for the Knoop Hardness Number. The results from the surface hardness and scraping test for 4 mm depths of cure are reviewed below. The 4 mm data was particularly significant in this study. The manufacturers' claims were based on polymerization of 4 mm increments and all data points started with 4 mm molds, therefore, all data is available for this thickness on all four composite materials.

For the Shade A2 group cured for 20 seconds at a thickness of 4 mm and assessed under the Knoop Hardness criteria, Grandio Flow and Revolution were the lowest and not statistically different. SureFil SDR Flow was found to be significantly greater than Grandio Flow and Revolution. Venus Bulk Fill was found to be significantly greater than all the other flowable materials tested in this group. According to the results in this data, Venus Bulk Fill exceeded the KHN ratio of 80%, with Surefil SDR Flow within the 80% threshold of complete polymerization when factoring in the standard deviation. See Figure 9.

For the shade A3 group cured for 20 seconds at a thickness of 4mm tested under the Knoop Hardness criteria, Grandio Flow and Revolution were the lowest and not significantly different from each other. SureFil SDR Flow was not significantly greater than Grandio Flow, but was significantly greater than Revolution and all materials were statistically lower than Venus Bulk Fill. Only Venus Bulk Fill exceeded the bottom/maximum KHN ratio of 80%. See Figure 9.

Grandio Flow and Revolution, A2 shade, cured for 40 seconds at a thickness of 4 mm utilizing the Knoop Hardness Tester was the lowest and not significantly different from each other. Grandio Flow and Revolution were significantly lower than SureFil SDR Flow and Venus, which were not significantly different from each other. SureFil SDR Flow and Venus Bulk Fill both exceeded the KHN ratio of 80%. See Figure 10.

The data compiled from the shade A3 group cured at 4 mm thickness for 40 seconds under the Knoop Hardness ratio found that statistically significant differences occurred between all four materials. Venus Bulk Fill, again, was the only composite material tested that exceeded the bottom/maximum KHN ratio of 80% in the group. See Figure 10.

The data compiled from the scrape technique or ISO 4049 indicated that for the shade A2, 20 second cure time group, Grandio Flow and Revolution were the lowest and not significantly different from each other, but significantly less than SureFil SDR Flow. However, Grandio Flow, Revolution and SureFil SDR Flow were significantly less than Venus Bulk Fill. See Figure 11.

For the group of materials in the shade of A3 cured for 20 seconds under the ISO 4049, all materials were significantly different statistically from each other. See Figure 11.

This was also true for the shade A2, 40-second cure groups which underwent the scrape technique. All materials were statistically different from each other. See Figure 12.

However, for the group of A3 shade, 40-second cure time utilizing the scrape technique, Revolution and Grandio Flow were not significantly different, and were the lowest, but significantly less than SureFil SDR Flow. However, Revolution, Grandio Flow, and SureFil SDR Flow were significantly less than Venus Bulk Fill. See Figure 12.

There are few studies comparing hardness tests and scrape technique. In this study, the depth of cure results achieved by the ISO 4049 and hardness test correlated well. Previous studies found that, although the scrape test appeared to overestimate depth of cure, there was a good correlation between both methods (Dewald, 1987, Moore 2008).   However, utilizing a linear regression analysis of the KHN profile data with a $R^2=0.77$, the depths of cure not only correlated but were similar in number. Some of the difference between the results in this study and prior studies may be due to the lack of criteria in assessing maximum hardness as well as the relevance of the Knoop Hardness ratios used in various studies to clinical practice.
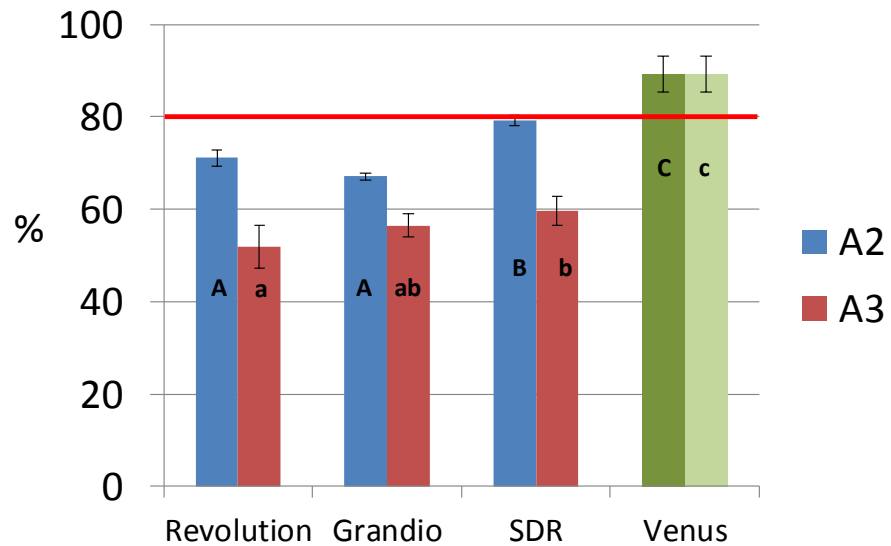
Percent Knoop Hardness Ratios

Figure 9: Bottom/maximum percent Knoop Hardness ratios for Shade A2 and A3 at 4 mm thickness with 20 seconds of curing time. Venus in Universal shade only. Same upper or lower case letters are not significantly different (p>0.05).
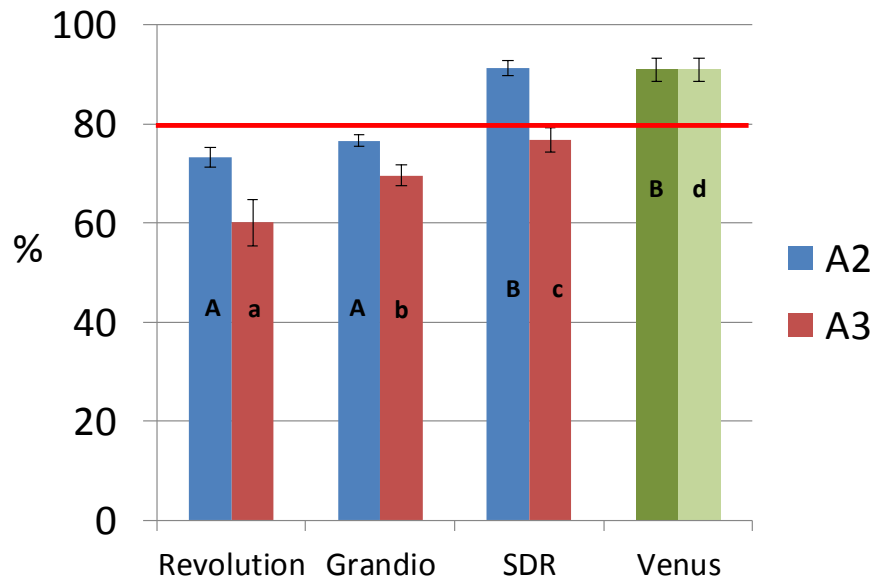

Figure 10: Bottom/maximum percent Knoop Hardness ratios for Shade A2 and A3 at 4 mm thickness with 40 seconds of curing time. Venus in Universal shade only. Same upper or lower case letters are not significantly different (p>0.05).

ISO 4049 Scrape Test

Figure 11: ISO 4049 scrape test for Shade A2 and A3 with 20 seconds of curing time. Venus in Universal shade only. Same upper or lower case letters are not significantly different ($p > 0.05$).
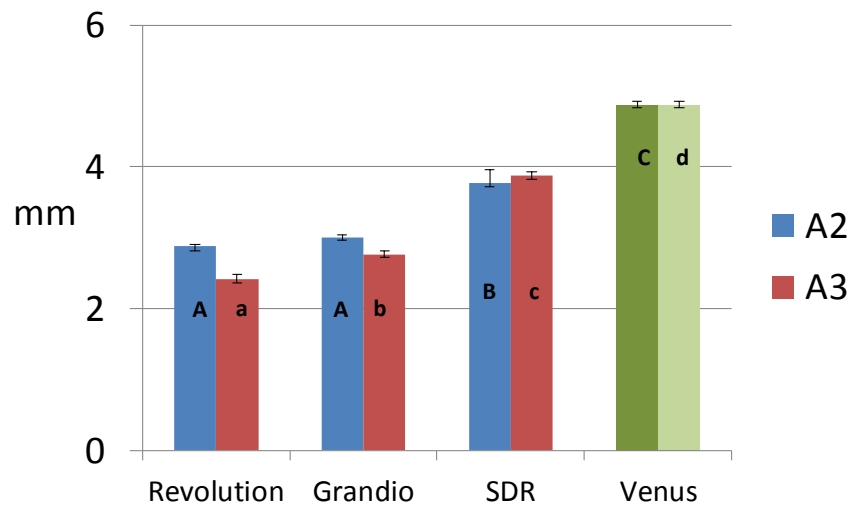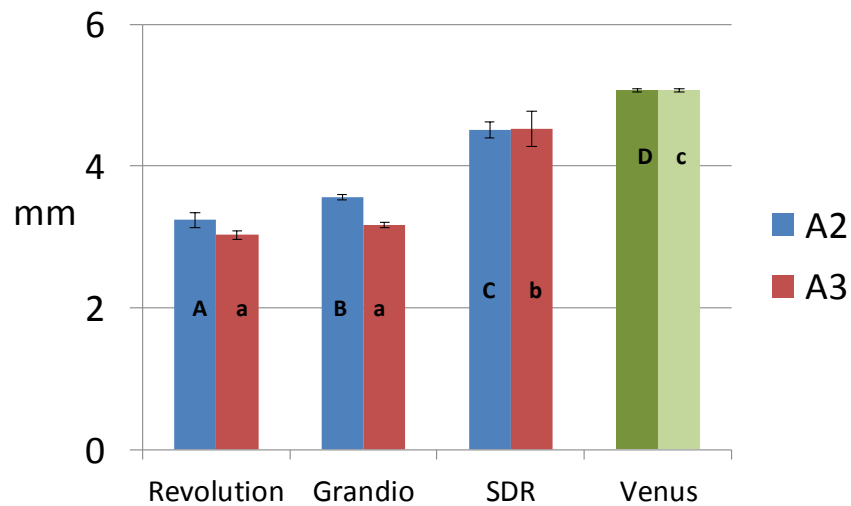

Figure 12: ISO 4049 scrape test for Shade A2 and A3 with 40 seconds of curing time. Venus in Universal shade only. Same upper or lower case letters are not significantly different ($p > 0.05$).

VI.   Conclusion:

Venus Bulk Fill predictably exceeded the manufacturer's claim of a 4-mm depth of cure using both Knoop Hardness Number ratios and the ISO 4049 scrape test at both 20 and 40 seconds of curing time. This study illustrated how shades can influence polymerization, with darker shades generally needing longer curing times to complete the polymerization process. Venus Bulk Fill currently has only a Universal shade which is very translucent compared to the other shades used in this study. The composite shades used in this study were those that were assessed to be the most commonly used by dentists. Therefore, the depths of cure data from this study are pertinent to the practice of dentistry. Grandio Flow did not demonstrate the depth of cure claimed by the manufacturer (4.3mm/40 sec and 3.5mm/20 sec). For shades A2 and A3, SureFil SDR Flow did not meet the manufacturer's claim of depth of cure of 4mm with a 20 second cure time. Although, with the standard deviation taken into account SureFil SDR Flow may have reached the KHN of 80% polymerization with shade A2.

Despite the universally excepted notion that the scrape test is often an overestimated test of depth of cure (DeWald, 1987; Moore, 2008), the data obtained and analyzed in this study found that, generally speaking, the ISO 4049 and the Knoop Hardness Ratio appeared to correlate well and demonstrated consistent results.

Overall, this study was a good parameter for depth of cure of the new flowable composite restorative materials. Curing time, shade selection and thickness of material all play a role in polymerization. Other factors that may be taken into account for future studies may also include curing distance. These new flowable composite materials with their new technology may have significantly improved properties than previous generations of flowable composites.

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